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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

JUN 20 1988

OPP OFFICIAL RECORD
HEALTH EFFECTS DIVISION
SCIENTIFIC DATA REVIEWS
EPA SERIES 361

OFFICE OF
PESTICIDES AND TOXIC SUBSTANCES

Subect: Revised Protocol (April 23, 1988) For Chlorine Generators Applied To Fresh Fruit, Submission of May 19, 1988 (No MRID No., RCB No. 3905).

From: Elizabeth T. Haeberer, Chemist Elizabeth T. Haeberer
Residue Chemistry Branch
Hazard Evaluation Division (TS-769C)

To: Walter Francis, PM Team 32
Disinfectants Branch
Registration Division (TS-767C)
and
Toxicology Branch
Hazard Evaluation Division (TS-769C)

Thru: Robert S. Quick, Head
Petition Review Section I
Residue Chemistry Branch
Hazard Evaluation Division (TS-769C)

In response to RCB's review of April 15, 1988 (Akin, Gump, Strauss et al submission dated 3/11/88. Review and Evaluation of Testing Protocol for Chlorine Generators Applied to Fresh Fruit, Elizabeth T. Haeberer), Frupac International Corporation has submitted a revised test protocol (April 23, 1988) for the use of chlorine generator pads to maintain freshness in table grapes during shipment and storage.

Conclusions

All of the questions and concerns raised in our review of May 19, 1988 have been addressed and answered satisfactorily. The revised test protocol appears adequate for the intended purpose of generating residue data.

Detailed Considerations

Exposure and Sampling

Questions raised in the April 15, 1988 review are listed below and followed by Frupac International Corporation's responses:

1. The total chlorine in grapes after chlorine gassing should be determined after exposure to 40 ppm concentration for 20 minutes rather than 20 ppm, since 40 ppm would be equivalent to the exposure proposed for commerce.

> Frupac accepts the proposed modification which appears in the revised protocol attached hereto. Please note that the 20 ppm level in the March 11, 1988 submission was a typographical error.

2. Grapes packed with pads containing calcium hypochlorite should be fumigated with chlorine gas at 40 ppm for 20 minutes prior to packing in order to simulate conditions proposed for commerce.

> Frupac accepts the proposed modification which appears in the revised protocol attached hereto. Please note that the proposed modification was intended to be incorporated in Section IV, Step 3 of the Handling and Packaging Procedures of the March 11, 1988 protocol submission.

3. Under "Sampling Procedures," item #4, samples also should be taken at 0 hours after removal of pad.

> Frupac accepts the proposed modification which appears in the revised protocol attached hereto. Please note that Section V, Step 3 of the March 11, 1988 submission had required samples at 0 hours after pad removal.

4. For comparison of total residues of grapes exposed to a single chlorine fumigation and grapes packed with pads, the grapes packed with pads should be fumigated with chlorine gas prior to packing with the pads. In addition, the samples should be stored at the same temperature and for the same length of time.

> As discussed with Residue Chemistry at the April 19 meeting, commercial use of a single chlorine gassing is not an intended application and is not the subject of this protocol. To the extent that the March 11 protocol was unclear on this point, please accept this clarification.

(This use question has been clarified. Grapes will be fumigated once with chlorine gas prior to packing with chlorine generator pads. Concurrently control experiments will be run with untreated grapes, and with grapes exposed to a single chlorine gas fumigation, but not packed with chlorine generator pads.)

5. Samples of bruised and shattered grapes must be analyzed also to determine whether a difference in residue concentration occurs.

Frupac accepts the proposed modification which appears in the revised protocol attached hereto.

6. Analytical method validation protocols should be included.

Frupac accepts the proposed modification which appears in the revised protocol, Appendices I and II attached hereto. Please note the abbreviated description of the validation procedure in Section IV of the March 11, 1988 submission.

The appropriate modifications, as described above, have been made in the subject protocol.

Analytical Methodology

The analytical methodology determines total chlorine residues, defined as below:

1. total organic chlorine
   a. total halogenated organics (TOX)
   b. trihalomethane (THM)
2. inorganic chloride

Samples are placed into pint Mason jars, sealed, and placed in dry ice for shipment to the laboratory, where they are stored at -20°C until processed. Sample jars are fitted with a blender blade assembly, placed at 0°C for 10 minutes, samples blended 2-5 seconds, and the homogenized samples immediately weighed into containers for analysis.

The THM content is determined using EPA Volatile Halocarbon Method 5020/8010. Samples are weighed into headspace sample vials, sealed and stored at -20°C until analysis. Residues are determined using GC equipped with a Hall detector.

The THM analysis methodology will be validated by spiking macerated control grapes with known quantities of chloroform, dibromochloromethane, and bromodichloromethane. The fortification level will be about 10 ppb. Four sets of fortification experiments will be conducted prior to initiation of the study. In addition, a recovery will be run every 10-15 samples.

Total halogenated organics (TOX) will be analyzed by weighing grape homogenate into a septum vial, adding hexane, shaking in a mechanical shaker, then processing in an ultrasonic water bath. The mixture is centrifuged, the hexane extract removed and analyzed by GC using a Hall detector as described in EPA Method 8010.

The TOX analytical method will be validated by fortification of macerated control grapes with known amounts of trihalomethane, and in addition, with a high boiling chlorinated organic compound such as heptachlor or chlordane. Several sets of recoveries will be run before the study is initiated. During the study a recovery will be run every 10-15 samples.

Analysis of inorganic chloride was described in the March 11, 1988 submission. Inorganic chloride will be determined using a chloride ion selective electrode, the electrode being placed directly into the macerated grapes. The anticipated detection level is in the ppm range.

cc: Haeberer, R. F., Circu, PP#7E3473, E. Eldredge (ISB/PMSD)
RDI: Robert S. Quick, 6/16/88; Richard D. Schmitt, 6/16/88
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